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PB-300 337

Evaluation of Stationary Source Particulate Measurement Methods Volume V. Secondary Lead Smelters

Battelle Columbus Labs, OH

Prepared for

Environmental Sciences Research Lab, Research Triangle Park, NC

Jun 79

United States
Environmental Protection
Agency

Environmental Sciences Research
Laboratory
Research Triangle Park NC 27711

EPA-600/2-79-116
June 1979

Research and Development

PB 300337

EPA

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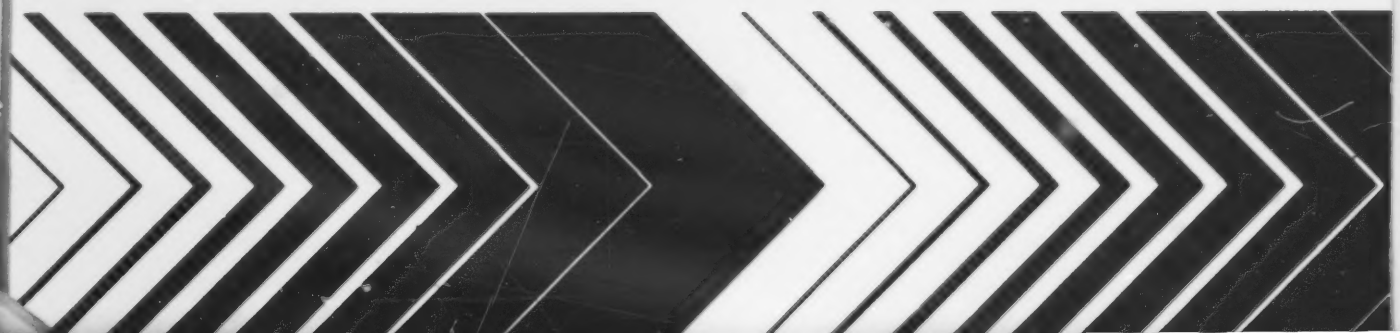

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TECHNICAL REPORT DATA
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1. REPORT NO. EPA-600/2-79-116	2.	3. RECIPIENT'S ACCESSION NO. PB300337
4. TITLE AND SUBTITLE EVALUATION OF STATIONARY SOURCE PARTICULATE MEASUREMENT METHODS Volume V. Secondary Lead Smelters		5. REPORT DATE June 1979 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) J. E. Howes, Jr., R. N. Pesut, and W. M. Henry		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Battelle, Columbus Laboratories 505 King Avenue Columbus, Ohio 43201		10. PROGRAM ELEMENT NO. 1AD712 (FY-75) 11. CONTRACT/GRANT NO. 68-02-0609
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Sciences Research Laboratory - RTP, NC Office of Research and Development U.S. Environmental Protection Agency Research Triangle Park, N.C. 27711		13. TYPE OF REPORT AND PERIOD COVERED Interim 10/73 - 6/77 14. SPONSORING AGENCY CODE EPA/600/09

15. SUPPLEMENTARY NOTES
Volume I was issued as EPA 650/2-75-051a, June 1975.
Volume II was issued as EPA 600/2-77-026, February 1977.

16. ABSTRACT

Operation of the Method 5 sampling system at secondary lead smelters with probe outlet and filter box temperatures of 93°C (200°F) and 149°C (300°F), respectively, yielded statistically equivalent mass loading results. Chemical analyses revealed no compositional differences in the particulate matter collected at the two different sampling temperatures. Sampling with MSA 1106 BH glass filters, as specified by Method 5, and with ADL quartz-type filters yielded no statistically significant differences for particulate mass loading or composition of the collected emissions. The Method 5 sampling train system did not induce compositional changes in the particulate collections. Samples taken from the probe and filter sections of the sampling train were compositionally similar to grab samples and to samples taken from the stack emission control baghouse collector.

The precision (repeatability) of particulate mass emission measurements by Method 5, on the basis of paired sampling tests, was about 1.5 percent when the two systems were operated simultaneously at a single fixed point in the stack. The collection efficiency of the system for lead compounds was very good.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
* Air pollution		13B
* Particles		21B
* Flue gases		14B
* Collection Methods		07B
* Evaluation		11F
* Temperature		
* Lead inorganic compounds		
* Smelters		

18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC	19. SECURITY CLASS (This Report) UNCLASSIFIED 20. SECURITY CLASS (This page) UNCLASSIFIED	21. NO. OF PAGES 40 22. PRICE PCA03 / MF A01

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

EPA-600/2-79-116
June 1979

EVALUATION OF STATIONARY SOURCE PARTICULATE MEASUREMENT METHODS
Volume V. Secondary Lead Smelters

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ABSTRACT

As part of an overall program to evaluate the EPA Method 5 procedure for measurement of particulate emissions as detailed in the Federal Register, Vol 36, No. 247, December 23, 1971, an experimental study was made of its specific applicability to secondary lead plant emissions. The study was carried out with two Method 5 sampling train systems operated simultaneously at a single point in the stack emission stream. A series of six statistically designed tests was conducted over a 5-day period to obtain data on the reliability of Method 5, the sensitivity of the method to variation of such key parameters as sampling system temperature, filter media, and particulate loading and to characterize the chemical composition of the emissions.

Comprehensive chemical analysos were made of particulates collected in the sampling system and from the baghouse control to ascertain if the sampling mode affected the composition of the particulate emissions. Essentially 100 percent of the particulates were accounted for by the chemical analyses. Compositional analysis of the gaseous species present in the stack gas stream also were performed. The results of the particulate and gas analyses do not indicate any chemical interactions with the sampling system components of the Method 5 train.

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1. INTRODUCTION

The Clean Air Act as amended in 1970 provides the impetus for programs to improve the air quality in the U.S. through research to broaden the understanding of the effects of air pollutants, research and development of techniques to control emissions, and the enactment of air quality regulations to protect the public welfare. Pursuant to Section 111 of the Act, the Environmental Protection Agency (EPA) on December 23, 1971, promulgated Standards of Performance for New Stationary Sources (amended) for fossil-fuel fired steam generators, incinerators, portland cement plants, and nitric and sulfuric acid plants[1]. On March 8, 1974, similar performance standards were issued for asphalt concrete plants, petroleum refineries, storage vessels for petroleum liquids, secondary lead smelters, secondary brass and bronze ingot production plants, iron and steel plants, and sewage treatment plants[2]. All new and modified sources in the preceding categories are required to demonstrate compliance with the standards of performance.

The performance standards are intended to reflect "the degree of emission limitation achievable through the application of the best system of emission reduction which (taking into account the cost of achieving such reduction) the Administrator determines has been adequately demonstrated"[3].

Compliance with required performance is determined by testing procedures specified with the standards. The use of the procedure called "Method 5 Determination of Particulate Emissions from Stationary Sources"[4] is specified in all instances where particulate mass emission measurements must be made. A copy of the Method as promulgated is given in Appendix A. The Method 5 procedure consists of isokinetic extraction of a sample from the emission stream with a heated probe and collection of the particulates on a heated filter. With the recent exception of fossil fuel-fired power plants[5], the same sampling system operating parameters have been adopted for all stationary sources.

The source categories subject to Method 5 particulate measurements include diverse processes which encompass a wide range of the following emission characteristics; moisture content, gas temperature, gas composition, particulate concentration and composition, and flow dynamics. Interaction of these emission properties with the Method 5 sampling technique can produce significant variations in the results of particulate emission measurements. The following are examples of some of the reactions which may affect particulate measurements.

(1) SO_3 or H_2SO_4 in emissions can condense to form sulfates which increase the mass of collected "particulates". The SO_3-H_2SO_4 dew point is dependent on SO_3 concentration and moisture content of the emissions.

(2) The filter particulate catch may present a surface for reactions with gaseous emission components such as SO_x and NO_x. Reactivity would be dependent on particulate loading and composition and on gas composition of the emissions as well as interactions with the filter media.

(3) Changes in gas temperature in the sampling system may alter the apparent particulate concentration through condensation, or volatilization.

Such interactions with the sampling process must be recognized and controlled if Method 5 is expected to yield reliable particulate measurements for individual source categories.

The work presented in this report was performed as a part of an EPA program to study the applicability of the Method 5 procedure to measurement of particulate emissions from a variety of stationary sources. Specifically, this work addresses the question of whether Method 5 provides an accurate, reliable measurement of particulate emissions from secondary lead smelters. Volumes I, II, and IV in this series cover similar studies of cement plants, oil-fired steam generators, and basic oxygen steel making furnaces, respectively. Volume III is on gas temperature control during Method 5 sampling.

2. CONCLUSIONS

The following conclusions regarding the methodology for determining particulate emissions from secondary lead plants may be drawn from this study.

SAMPLING SYSTEM TEMPERATURE

Operation of the Method 5 sampling system with probe outlet and filter box temperatures of 93°C (200°F) and 149°C (300°F) yielded equivalent mass loading results based on statistical differences in the particulates collected at the two different sampling system temperatures. Therefore, it is concluded that operation of the sampling system at the minimum temperatures recommended in Method 5 produces representative mass emission measurements and that variations from 93 to 149°C do not affect the results.

FILTER MEDIA

Sampling with MSA 1106 BH glass filter as specified by Method 5 and the ADL quartz-type filter yielded no statistically significant differences either in respect to particulate mass loading or in the compositions of the collected emissions.

CHEMICAL INTERACTIONS

The Method 5 sampling train system did not induce compositional changes in the particulate collections. Samples taken from the probe and filter sections of the sampling train were compared compositionally with grab samples and with samples taken from the stack emission control baghouse collector and were found to be similar in chemical compositions.

PRECISION

The precision (repeatability) of particulate mass emissions by Method 5 on the basis of paired sampling tests was found to be about 1.5 percent when the two systems were operated simultaneously at a single fixed point in the stack. This precision was attained over a 5-day sampling period despite considerable variation in the particulate emission loadings.

COLLECTION EFFICIENCY

Examination of the impinger collections revealed only a very small fraction (0.4 percent of the Pb -- which constituted the probe and filter

section loadings was transported through the filter. Approximately 26 percent of the total As passed through the filter and was collected in the impingers.

PARTICULATE EMISSION COMPOSITIONS

The major components of the particulate emissions was found to be lead, probably as $PbSO_4$ and/or $PbCl_2$ which constituted 80 to 85 percent of the emissions. Other heavy metals were Sn, Cd, Zn, Sb, and As, again probably present as chlorides and/or sulfates and in quantities of about 4, 1, 1, 0.5, and 0.5 percent of the particulate emissions. Organics, as indicated by the carbon contents of the emissions and by the extracts from the impinger solutions, constitute less than one percent by weight of the emissions and these are comprised mostly of relatively low molecular weight aliphatic compounds, carbonyls, esters, and diacids.

3. RECOMMENDATIONS

The results of this study indicate the EPA Method 5 is a satisfactory procedure for measuring particulate emissions from secondary lead plants and that no modifications are required to obtain representative and reproducible mass emission measurements.

4. OBJECTIVES

The objective of the overall EPA program is the evaluation of the applicability and reliability of Method 5 (conducted as specified in the Federal Register, December 23, 1971) for the determination of particulate mass emissions from stationary sources for which performance standards have been promulgated. The portion of the overall program covered by this report is aimed at evaluation of Method 5 performance when the procedure is applied to secondary lead smelters. The study sought to identify any characteristics of the sampling method or unique properties of process emissions which would adversely effect particulate measurements and, if possible, recommend appropriate corrective measures in sampling methodology.

A secondary objective in this program is the characterization of the emission species particularly in regard to heavy metal concentrations.

5. EXPERIMENTAL WORK AND RESULTS

EXPERIMENTAL APPROACH

An experimental plan was drawn up to test and evaluate the sensitivity of Method 5 to key sampling variables, including temperature, filter media, loading and to determine the specific chemical composition of the particulate emissions. The approach used was similar to that employed previously[6] and consisted of concurrent sampling at a single point in the stack with two Method 5 sampling train systems operated under the chosen conditions of study. Experiments were carried out in a statistically designed test pattern to permit the significance of observed differences to be assessed. The Method 5 sampling procedure as detailed in the Federal Register was strictly adhered to in the experimental tests except for stack profiling and, of course, the use of the planned variations designed to test the sensitivity of the method and to reveal potential problem areas.

Filter and probe collections were analyzed gravimetrically for mass loadings and in detail chemically to detect changes, if any, induced by the collection process. The sampling catches were compared compositionally with grab samples and with collections from the baghouse.

Descriptions of the secondary lead emission source, experimental testing, and test results are detailed in the following sections.

PROCESS AND SAMPLING SITE DESCRIPTIONS

Secondary Lead Smelting Process

This experimental study was performed at a secondary lead plant which uses a blast furnace for the smelting and refining process. The furnace is fed nearly continuously with coke, cast iron scrap, batteries, limestone, a silaceous slag, drosses and other lead-containing residues. About 10 charges are fed each 8-hour shift with a 7100 pound charge yielding about 4800 pounds of lead. Figure 1 shows the gas flow through the plant. Air is blown into the furnace to burn the coke in the feed. The heat of combustion melts the lead and the coke reduces the lead oxides. The off-gas from the furnace is combusted in an afterburner to oxidize any odiferous compounds and to incinerate oily and sticky materials which may blind the fabric filters. The off-gas is cooled in three air-cooled cyclones in series which also remove most of the dust. The remaining dust is removed in a baghouse. The gas is then exhausted through a 154-ft high stack.

A sketch of the blast furnace is shown in Figure 2. The furnace, rated at about 77 tons/day, is loaded from the top and tapped for lead recovery at the bottom of the hearth. Slag, which normally floats on the surface of the lead, also is drawn off near the bottom of the furnace. The

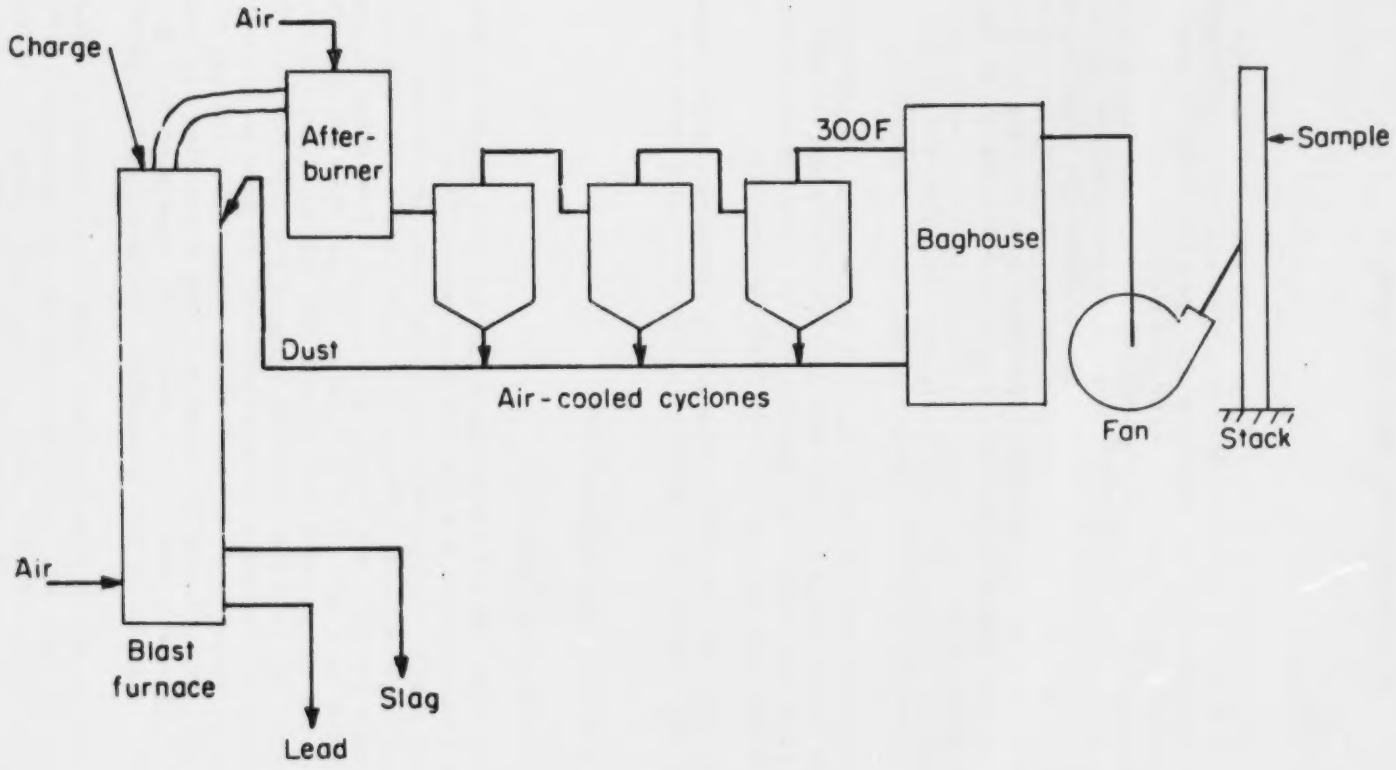

Figure 1. Gas flow diagram blast furnace secondary lead smelter.

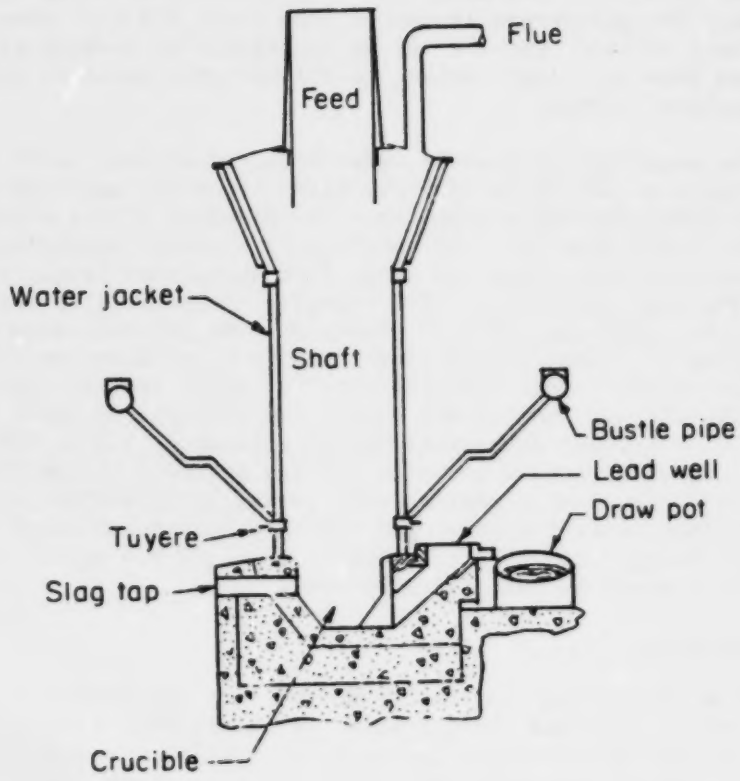

Figure 2. Lead blast furnace.

main gas stream leaving the blast furnace is heated to about 650 C (1200 F) in an afterburner with a natural gas flame in which most of the hydrocarbons and some CO in the exhaust gas are burned. The gas then passes through three forced air and water-cooled cyclones which also remove some particulates. The gas stream is cooled from about 650 C to about 150 C in the cyclones by heat transfer and by dilution with leakage air. Each volume of gas from the blast furnace is diluted with about 11 volumes of air in the exhaust system.

The sampling experiments were conducted at roof level (about 34 ft above ground) on the 46.9m (154-ft) stack after the gas from the blast furnace had passed through a baghouse. The diameter of the stack was about 1.22 m (4 ft) with two 7.62 cm (3-inch) diameter portholes, set at 90 degrees from each other and about 4 ft above roof level, providing access for the sampling probes. The velocity, pressure drop and temperature profiles of the stack are shown in Figure 3. The general emission characteristics of blast furnace stack emissions are given on Table 1. With the high volume of air dilution into the stack the gas composition is essentially that of ambient air except for the CO_2 and small amounts of SO_2 and CO. Particulate content varied from about 134 to 378 mg/Nm^3 during the test runs carried out over a 5 day period. During the 5 day sampling period the plant operators were having considerable difficulty with the emission control system, and the particulate emissions varied considerably, being well above the highest level of 6.4 mg/Nm^3 obtained in 1972 at this plant by Battelle personnel.

Sampling Equipment

Particulate sampling was performed with two identical Method 5 sampling trains operated concurrently. A single Type S pitot tube positioned equidistance between the sampling nozzles was used to measure velocity pressure of the stack gas. With the exceptions listed below, the trains consisted of components assembled as described in the Federal Register, Sections 2 and 3 of the Method 5 procedure.

Temperature Measurements

The glass connectors from the probe outlet to the filter and the filter outlet to the first impinger were modified to permit additional measurements of gas sample temperature. The probe outlet-to-filterholder connector contained a thin-wall thermocouple well which extends about 5.1 cm (2 inches) into the outlet end of the probe. The filterholder-to-impinger connector was fitted with a bi-metal dial thermometer. The tip of the thermometer was positioned about 1.3 cm (0.5 inch) from the filterholder frit.

Filter Materials

Mine Safety Appliance (MSA) 1106 BH glass fiber filter materials and Arthur D. Little experimental quartz filters were used throughout the test series.

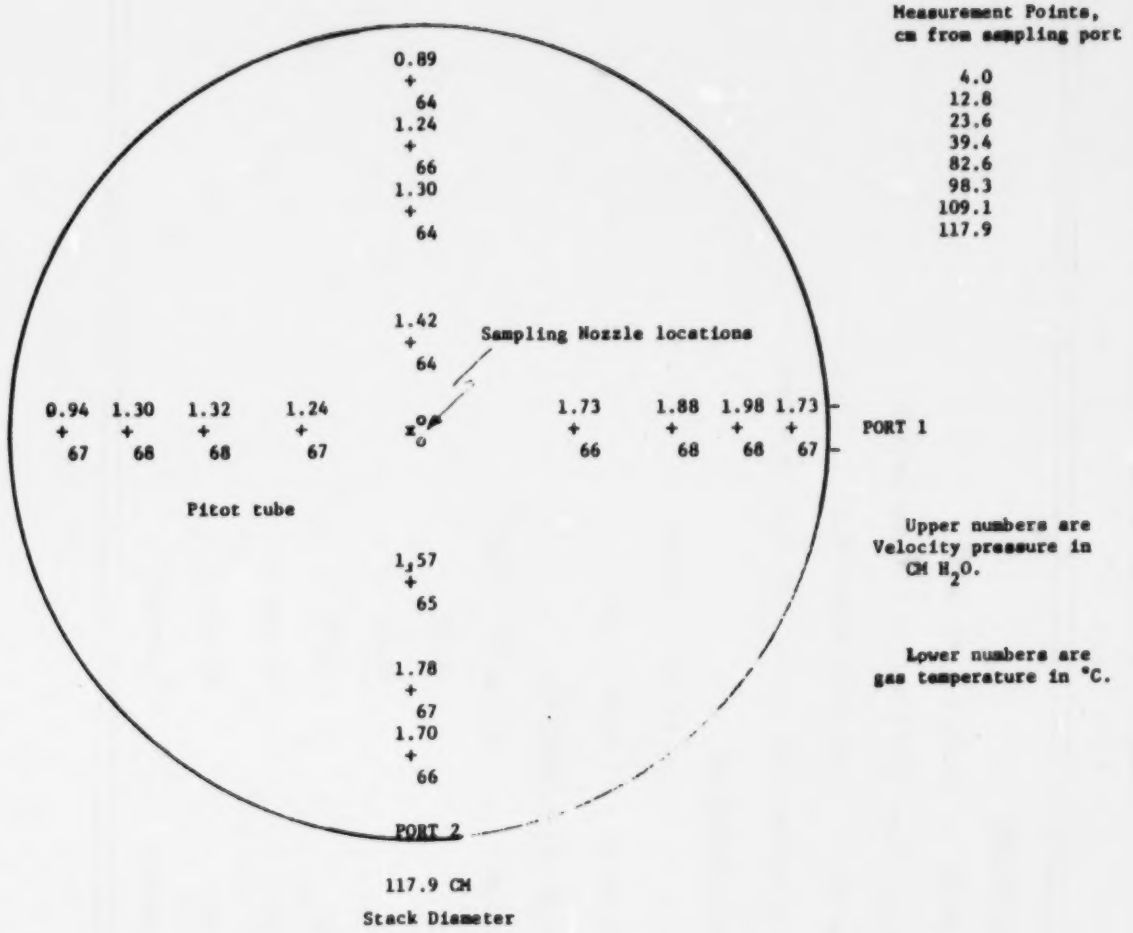

Figure 3. Velocity pressure and temperature profile of stack.

TABLE 1. EMISSION SOURCE CHARACTERISTICS

Flue Gas Conditions	
Temperature	61-74 C (142-165F)
Volumetric flow	49277 Nm^3/hr (29,000 DSCFM)
Average ΔP	1.47 cm (.58 in) H_2O
Static pressure	0.76 cm (0.3 in) H_2O, negative
Flue and Port Dimensions	
Flue size -	1.22m (4 ft) diameter
Port diameter -	7.62cm (3 in)
Composition of Stack Emissions	
Particulates	∿216 mg/Nm^3 (avg.)
N_2	78.5%
O_2	18.5%
CO_2	2.3%
CO	65 ppm
SO_2	300 ppm
Moisture	∿3 percent

Impingers

In some runs, the first impinger was loaded with 6N HNO_3 and the second with 1N NaOH to collect any volatile metals which might pass through the box filter.

Gas analyses for CO_2 and O_2 were performed with Fyrite equipment. In addition grab samples were taken with an evacuated glass gas sampling bulbs for mass spectrometric analysis of gases and low molecular weight organic species.

SAMPLE COLLECTION AND ANALYSIS PROCEDURES

Particulate Sampling

In all tests, particulate sampling was performed concurrently with two identical sampling train units (designated A and B) each with a separate operator. Sampling system equipment and operating conditions used in the tests were varied in accordance with a statistically designed experimental pattern.

All sampling was performed at a fixed-point at the center of the duct in an area of nearly uniform velocity. Sampling probes of the two systems were inserted into the duct through two ports, situated in the stack at a 90-degree angle to each other, so that the pitot tube attached to one of the probes was positioned equidistance between the sampling nozzles. The separation between the pitot tube and each nozzle was about 2.5 cm (1 inch).

At the start of each test day, the laboratory calibration of the gas metering components of both sampling systems was checked by setting the orifice manometer (ΔH) to the meter box calibration factor ($\Delta H@$) and measuring the flow rate through the dry gas meter over a 5-minute period. A flow rate of 0.021 m^3/min (0.75 cfm) confirmed that the gas metering system remained in calibration.

The preparation of the particulate collection trains for all tests was performed as specified in Paragraph 4.1.2 of Method 5.

In performance of the tests, sampling trains were operated as described in Paragraph 4.1.3 of Method 5 with the exception that readings of ΔP, ΔH, stack temperature and sampling system temperatures were recorded at 10-minute intervals. The velocity head (ΔP) for both systems was determined from one pitot tube and nomographs were used to obtain the proper sampling rate (ΔH). Temperature measurements were obtained at the points shown in Figure 4.

The sampling period for each test was 150 minutes and the total dry gas sample volumes at isokinetic sampling rates ranged from about 3 to 5.5Nm^3.

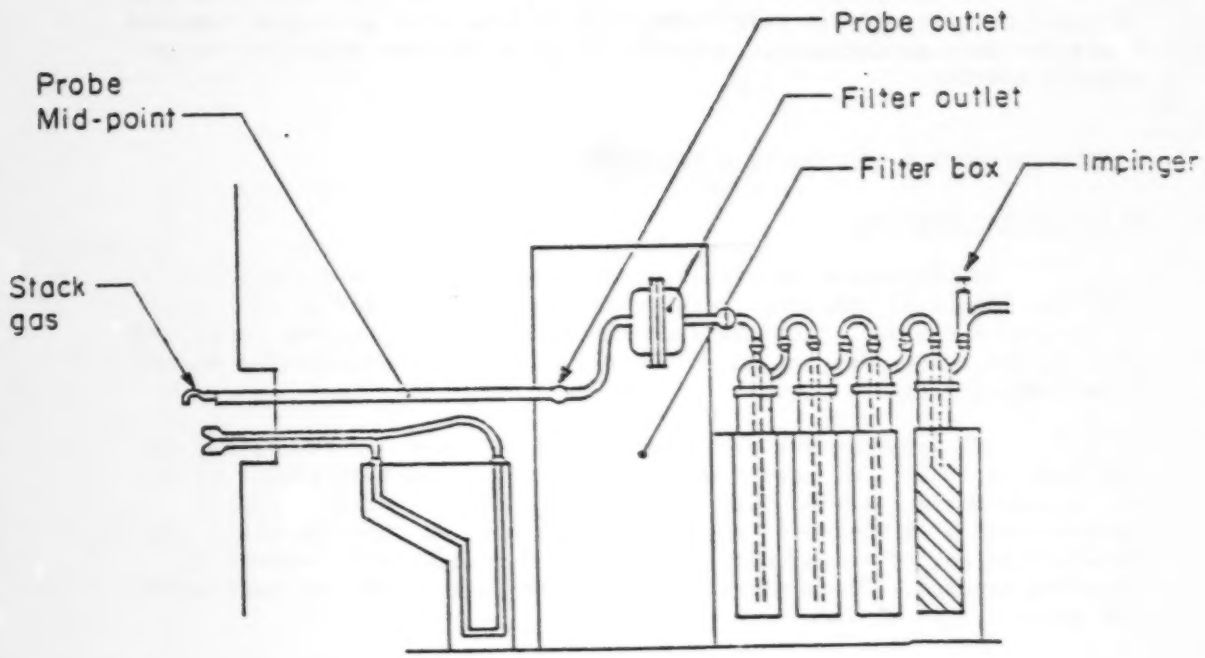

Figure 4. Temperature measurement points.

After completion of the tests, the trains were again leak checked, sealed to prevent contamination, and transferred to the sample recovery area.

Sample Recovery and Analysis

Filters were removed from holders, sealed in Petri dishes and immediately placed in a desiccator. The probe and nozzle were disassembled with washed separately. First, the probe was first rinsed with acetone without brushing, then rinsed with acetone while slowly inserting and removing a Nylon brush in a rotating fashion. The acetone wash and brushing were continued until visual inspection indicated that all particulates were removed. The brush was thoroughly flushed with acetone prior to removal from the probe. The probe wash (usually about 100 to 150 ml) was collected in an Erlenmeyer flask sealed onto the probe outlet ball joint. Particulates were recovered from the nozzle and the inlet half of the filter holder by alternately brushing and rinsing with acetone. The wash solutions from all three components (probe, nozzle, and filterholder) were combined for analysis.

At least one 200 ml acetone blank was obtained each day from the wash bottle dispenser. All acetone wash solutions and blanks were stored in glass bottles with Teflon-lined caps for transfer to the laboratory for analysis.

The filters and particulate catch were desiccated at least 24 hours (usually longer) prior to weighing. Acetone wash solutions were evaporated to dryness in a reverse airflow, clean hood and the residues were desiccated to a constant weight (usually 24 to 48 hours). Residues and filters were weighed to the nearest 0.1 mg.

All calculations were performed as described in Section 6 of Method 5.

TEST DESCRIPTIONS AND RESULTS

Variables selected for study were the sampling system temperature, the filter media, and the particulate loading. These, together with detailed chemical analyses of the particulate catches and gaseous emission to indicate potential chemical interactions (and possible formation of pseudo particulates) should indicate the reliability and sensitivity of Method 5 to the measurement of emissions from secondary lead plant operations.

The randomized test pattern for the study of the effects of the system variables temperature and filter media is shown in Table 2. An additional test pair was run wherein the filter in one system was changed midway through the 4-hour test period to assess the effect of particulate loading.

The experimental tests were carried out over a 5-day period when the plant was operating at full capacity. Some difficulty was being encountered with the emission control equipment during this period which resulted in overall higher and more erratic stack emissions than normally anticipated.

TABLE 2. RANDOMIZED TEST PATTERN FOR STUDY OF EFFECTS OF SYSTEM TEMPERATURE - FILTER MEDIA - SECONDARY LEAD PLANT DATA

Replication	Block	Test Number	System A[a]		System B[a]	
			Temperature, C	Media	Temperature, C	Media
1	1	1[b]	149	ADL	149	MSA
1	2	2[b]	93	ADL	93	MSA
2	1	3[c]	149	ADL	93	MSA
2	2	4[c]	149	MSA	93	ADL
3	1	5[d]	93	ADL	149	ADL
3	2	6[d]	149	MSA	93	MSA

(a) Both System A and System B are Method 5 trains.
(b) Tests 1 and 2 confound temperature with Blocks.
(c) Tests 3 and 4 confound temperature/filter with Blocks.
(d) Tests 5 and 6 confound filter with Blocks.

Particulate Measurements

The particulate collection data obtained in the seven paired runs are given in Table 3. Sampling and stack gas data for the runs are presented in the Appendix B.

The analysis of variance of the six runs (1 through 6) to study filter and temperature effects is shown in Table 4. The conclusions drawn from the statistical analysis are that neither variation of the filter media (MSA and ADL) nor the sampling system temperature (at 93 and 149C) had a statistically significance on the particulate mass results. Analysis of the variation in the six test pairs shows good repeatability with an overall coefficient of variation of 1.48 percent.

In Run 7, the System B box filter was changed midway through the four hour sampling period. System A filter was unchanged. The good agreement in the pair of results indicates that there is not a significant effect of mass loading or of reaction of the stack gases with the particulates catch on the filter.

Compositional Analyses

Probe and Filter Residues. Selected particulate catches from the probe, filter, and impinger segments of the sampling train, together with a bulk sample and a sample taken from the baghouse, were analyzed in detail to ascertain the compositions of the emissions.

Metallic elements were analyzed semiquantitatively by optical emission spectrography and the results obtained are shown in Table 5. These data indicate no great differences among the probe, filter catch, baghouse, or grab sample compositions. Selected additional probe and filter catch samples together with the grab sample and a baghouse sample were analyzed quantitatively and the results obtained are shown in Table 6. Again no major differences are observed among these samples. From averages of the quantitative data from Table 6 the cation, anion, and C values total 96.2 percent with presumably additional oxygen in the form of metallic oxides making up the difference between 96.2 and 100 percent. Cation-anion ratio given in Table 7 show an imbalance of 0.171 (0.913 - 0.742 = 0.171) which if attributed to oxygen gives a content of 1.4 percent. With the value for the additional undetermined oxygen and the Table 5 average values for cations, anions, and carbon compositional balance of 97.6 percent is obtained. This value, within analytical error, is sufficiently close to 100 percent to indicate that no other elements are present in more than trace amounts.

Impinger Residues. Total impinger catches (extracted organics plus aqueous residue) averaged approximately 7 percent of the front half particulate catch. The impinger catches collected in 6N HNO_3 and 1N NaOH were examined analytically to determine if potentially volatile metallic elements such as Pb and As, were carried through the filter. Organic extracts were analyzed gravimetrically and by infrared spectrometry.

TABLE 3. PARTICULATE COLLECTION DATA - SECONDARY LEAD PLANT

Run No	Temperature C		Filter	Particulate Catch, mg			Particulate Loading, mg/Nm^3
	Filter	Probe Out		Filter	Probe	Total	
1A	142	148	ADL	766.1	344.5	1110.6	209.0
B	149	148	MSA	732.9	298.8	1031.7	203.8
2A	94	95	ADL	305.6	160.8	556.4	159.3
B	97	93	MSA	390.9	138.8	529.7	158.9
3A	92	94	ADL	488.7	125.3	614.0	180.5
B	149	148	MSA	459.9	135.6	595.5	182.5
4A	94	94	MSA	377.8	90.2	468.0	133.6
B	149	148	ADL	354.3	99.3	453.6	134.4
5A	148	148	MSA	414.0	177.5	591.5	170.0
B	96	99	MSA	397.8	178.9	557.7	166.2
6A	92	92	ADL	949.6	325.9	1275.5	377.8
B	151	148	ADL	972.1	232.0	1204.1	369.8
7A (a)	123	122	ADL	691.1	165.1	856.2	290.6
B	118	120	ADL	693.6	140.3	833.9	293.9

(a) Filter changed midway through 4 hour test.

TABLE 4. ANALYSIS OF VARIANCE - FILTER MEDIA/SAMPLING SYSTEM TEMPERATURE EXPERIMENTS - SECONDARY LEAD PLANT

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio[a]	Conclusion
Filter Media (F)	1	2.4200	2.4200	0.1566	Not Significant
Temperature (T)	1	0.2450	0.2450	0.0159	Not Significant
F x T	1	6.1250	6.1250	0.3964	Not Significant
Reps	2	28,291.3067	14,145.6533	--	--
Blocks/Reps	3	46,806.0300	15,602.0100	--	--
Remainder	3	46.3500	15.4500	--	--
Total	11	75,152.4767	6832.0433	--	--

(a) For an F-ratio with 1 to 3 degrees of freedom, any calculated value of F exceeding 5.54 is significant at the 90 percent confidence level, exceeding 10.13 is significant at the 95 percent confidence level, and exceeding 34.12 is significant at the 99 percent confidence.

TABLE 5. ANALYSIS OF PARTICULATE EMISSIONS FROM SECONDARY LEAD SMELTER[a]

Sample	Weight Percent of Element in Sample																						
	Pb	Sn	Zn	As	Na	K	Si	Fe	Sb	Cu	Cd	Mg	Mn	Bi	Cr	Al	V	Ni	Ca	Ba	B	Mo	Ag
Filter (3A)	40-60	1-3	0.3	0.3	2-4	1	0.2	0.1	0.2	0.03	0.5-1	≤0.001	0.003	0.003	≤0.001	0.003	≤0.001	≤0.001	0.03	≤0.001	≤0.001	≤0.001	≤0.0005
Filter (3B)	40-60	1-3	0.3	0.3	2-4	1	0.2	0.1	0.2	0.03	0.5-1	≤0.001	0.001	0.003	≤0.001	0.003	≤0.001	≤0.001	0.03	≤0.001	≤0.001	≤0.001	≤0.0005
Probe (3A)	40-60	1-3	0.3	0.2	2-4	1	0.5	0.3	0.2	0.03	0.5-1	0.01	0.005	0.003	0.005	0.2	≤0.001	0.003	0.03	≤0.001	≤0.001	0.003	≤0.0005
Probe (3B)	40-60	1-3	0.3	0.2	2-4	1	0.3	0.3	0.2	0.03	0.5-1	0.01	0.005	0.003	0.005	0.2	≤0.001	0.003	0.03	≤0.001	≤0.001	0.003	≤0.0005
Baghouse catch	40-60	1-3	0.3	0.1	2-4	1	0.02	0.3	0.2	0.03	0.5-1	≤0.001	0.001	0.003	0.001	≤0.001	≤0.001	≤0.001	0.02	≤0.001	≤0.001	≤0.001	≤0.0005
Bulk sample	40-60	1-3	0.3	0.2	2-4	1	0.02	0.3	0.2	0.03	0.5-1	≤0.001	0.002	0.003	0.001	.003	≤0.001	≤0.001	0.02	≤0.001	≤0.001	≤0.001	≤0.0005

(a) Analysis performed by optical emission spectroscopy

TABLE 6. CHEMICAL ANALYSIS OF PARTICULATE EMISSIONS FROM SECONDARY LEAD SMELTING PROCESS

Sample	Weight Percent in Sample												
	Pb	Sn	As	Cd	Sb	Zn	$SO_4^=$	Cl^-	HCO_3^-	C	N	P	pH
Method 5 Filter (1A)	59.5	2.09	0.20	0.62	0.21	0.52	7.53	19.3	ND	.15	—	0.05	4.4
Method 5 Filter (2B)	60.1	2.11	0.24	0.64	0.31	0.53	4.61	23.4	1.0	--	--	0.07	5.4
Method 5 Probe Residue (1A)	55.1	2.07	0.18	0.75	0.22	0.59	6.50	20.7	0.22	1.7	--	0.06	4.8
Method 5 Probe Residue (2B)	55.7	2.27	0.20	0.55	0.22	0.70	6.09	21.6	0.43	--	--	0.06	5.2
Grab Sample of Stack emissions	60.5	2.05	0.22	0.64	0.27	0.47	6.92			.34	.21	0.07	4.7
Baghouse Catch	61.2	1.97	0.22	0.66	0.25	0.46	5.94	22.4	0.44	--	--	0.06	5.1

TABLE 7. CATION/ANION BALANCE IN SECONDARY LEAD PLANT EMISSION SAMPLES

Cation	Percent	Equivalent	Anion	Percent	Equivalent
$Pb^{=}$	58.7	0.567	$SO_4^{=}$	6.20	0.129
$Sn^{=}$	2.1	0.071	Cl^{-}	21.5	0.606
$Zn^{=}$	0.55	0.017	HCO_3^{-}	0.4	0.007
$Cd^{=}$	0.65	0.012	Totals	28.1	0.742
$Sb^{\equiv}$	0.25	0.006	Total cation = 0.913		
$As^{\equiv}$	0.21	0.008	Total anions = 0.742		
Na^{-}	3.0	0.13	Difference = 0.171: calculated as O_2 = 1.4 percent		
K^{-}	1.0	0.026			
$Si^{\equiv}$	0.2	0.040			
$Ca^{=}$	0.05	0.003			
$Fe^{\equiv}$	0.2	0.011			
$Al^{\equiv}$	0.2	0.022			
Totals	67.11	0.913			

Organic constituents were determined from chloroform-ether and acetone extracts from selected impinger washes. By weight these constituted about 9 to 15 percent, respectively, of the total residues in the impinger washes. Infrared spectrometric analysis showed the chloroform-ether extracts to be principally a complex mixture of carbonyl components with relatively short chains (aliphatics - high CH_3), a small amount of aromatic structure, some ether structure, and a small amount of hydroxyl. The acetone extract primarily consisted of sulfonic acids.

Inorganic analysis of the HNO_3 and NaOH impinger solutions were made by optical emission spectrography. The results show that about 0.4 of the total lead was collected in the impingers. However, about 26 percent of the total arsenic passed through the filter and was collected in the impinger.

Gaseous Components

Grab samples were taken of the stack gases by use of evacuated sampling bulbs and these were analyzed by gas chromatography and gas mass spectrometry. The results obtained are given in Table 8.

Except for the hydrocarbons found in Sample 1, the following were not detected and were less than 2 ppm in both samples: C_2H_2, C_2H_4, C_2H_6, C_2H_8, C_4H_{10}, C_5H_{10}, C_6H_{14}, CH_3OH, C_2H_5OH, COS, CS_2, H_2S and HCl. As stated previously, the principal component of the highly diluted stack gas in air. An SO_2 level of 200 to 400 ppm was found plus about 65 ppm of CO. No HCl nor free chorine were detected.

TABLE 8. GAS CHROMATOGRAPHIC AND MASS SPECTROMETRIC ANALYSIS OF GASEOUS EMISSIONS FROM SECONDARY LEAD SMELTER

Sample	Volume percent										
	CO_2	O_2	N_2	A	H_2	SO_2	C_6H_4	C_4H_{10}	C_3H_8	CO	CH_4
1	2.3	18.1	78.5	0.94	0.06	0.04	0.02	0.04	0.02	62	4
2	2.4	18.8	78.5	0.94	0.06	0.02	<0.01	<0.01	0.01	67	4

6. DISCUSSION

The parameters selected for study - sampling system temperature, filter media, and filter loading -- were those deemed most likely to have an effect in the application of Method 5 to the determination of particulate mass emissions from secondary lead plants. However, results of this study indicate that these factors within the limits investigated to not significantly effect the mass emission results obtained with Method 5. Unfortunately, during the sampling period, emissions from the secondary lead plant studied were higher than normal. Therefore the study results must be qualified somewhat by this fact. For example, at a lower particulate loading, the effects of the reaction of SO_2 or SO_3 with the filter medium could result in a detectable error.

The chemical analysis provide important data which corraborate the findings of the particulate mass measurements. The probe and filter catches have essentially the same composition as a grab sample and a sample of the baghouse catch which also indicates that the particulates collected by Method 5 are representative of the emission source and that no alterations occur during sampling.

7. REFERENCES

1. Federal Register, Volume 26, No. 247, pgs 24876-24895, Thursday, December 23, 1971.

2. Federal Register, Volume 30, No. 47, pgs 9308-9323, Friday, March 8, 1974.

3. Federal Register, Volume 30, No. 177, pg 32852, Wednesday, September 11, 1974.

4. Reference 1, pgs 24888-24890.

5. Reference 3, page 32856.

6. Evaluation of Stationary Source Particulate Measurement Methods -- Volume 1, Portland Cement Plants, EPA-650/2-75-051a, June 1975.

7. Sittig, M., Environmental Sources and Emissions Handbook, Noyes Data Corp., Park Ridge, New Jersey, pp 331-333 (1975).

APPENDIX A

EPA METHOD 5
Federal Register, December 23, 1971

2.1.4 Filter Holder—Pyrex[1] glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers / Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F, dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to ±0.1 mg.

2.3.4 Trip balance—500 g capacity, to measure to ±0.05 g.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH[1], or equivalent, numbered for identification and preweighed.

3.1.2 Silica gel—Indicating type, 6–16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

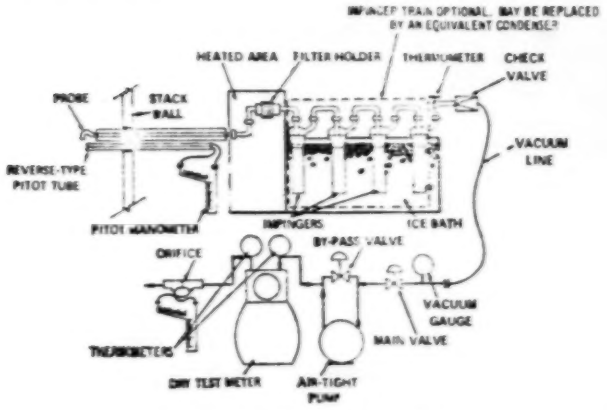

Figure 5-1. Particulate-sampling train.

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METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and its weight is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling train. The design specifications of the particulate sampling train used by EPA (Figure 5-1; are described in APTD-0581. Commercial models of this train are available.

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system capable of maintaining a minimum gas temperature of 250° F. at the exit end during sampling to prevent condensation from occurring. When length limitations (greater than about 8 ft.) are encountered at temperatures less than 600° F. Incoloy 825[1], or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 600° F must have been approved by the Administrator.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

[1] Trade name.

2.1.4 Filter Holder—Pyrex[1] glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers / Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F, dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to ±0.1 mg.

2.3.4 Trip balance—500 g capacity, to measure to ±0.05 g.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH[1], or equivalent, numbered for identification and preweighed.

3.1.2 Silica gel—Indicating type, 6–16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

3.3.2 Desiccant—Drierite[1] indicating.

4. *Procedure.*

4.1 Sampling.

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Weigh to the nearest gram approximately 300 g. of silica gel. Label a filter of proper diameter, desiccate[2] for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50%. Place 100 ml. of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g. of preweighed silica gel in the fourth impinger. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging up the inlet to the filter holder and pulling a 15 in. Hg vacuum. A leakage rate not in excess of 0.01 c.f.m. at a vacuum of 15 in. Hg is acceptable. Attach the probe and adjust the heater to provide a gas temperature of about 250° F. at the probe outlet. Turn on the filter heating system. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 70° F., or less. Temperatures above 70° F. may result in damage to the dry gas meter from either moisture condensation or excessive heat.

4.1.3 Particulate train operation. For each run, record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point at least every 5 minutes, and when significant change in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least 2 hours. At each traverse point, sampling time should be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without additional calculations. APTD-0576 details the procedure for using these nomographs. To run off the pump at the conclusion of each run and record the final reading. Remove the probe and nozzle from the stack and handle in accordance with the sample recovery process detailed in section 4.2.

[1] Trade name.

[2] Dry using Drierite[1] at 70° F ± 10° F.

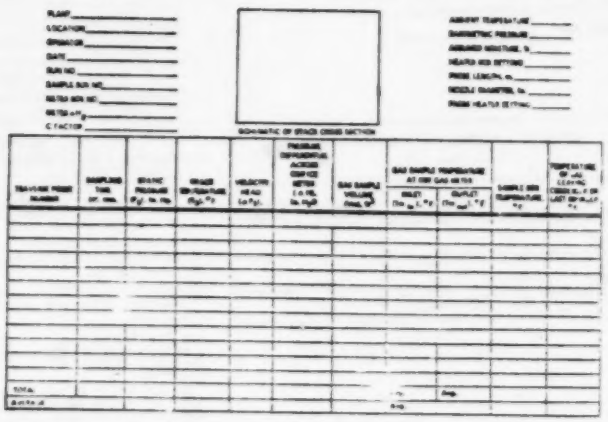

Figure 5-2. Particulate field data.

4.2 *Sample recovery.* Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. Measure the volume of water from the first three impingers, then discard. Place the samples in containers as follows:

Container No. 1. Remove the filter from its holder, place in this container and seal.

Container No. 2. Place loose particulate matter and acetone washings from all sample-exposed surfaces prior to the filter in this container and seal. Use a razor blade, brush, or rubber policeman to lose adhering particles.

Container No. 3. Transfer the silica gel from the fourth impinger to the original container and seal. Use a rubber policeman as an aid in removing silica gel from the impinger.

4.3 *Analysis.* Record the data required on the example sheet shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate, and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 2. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 3. Weigh the spent silica gel and report to the nearest gram.

5. *Calibration.*

Use methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater. Recalibrate after each test series.

6. *Calculations.*

6.1 *Average dry gas meter temperature and average orifice pressure drop.* See data sheet (Figure 5-2).

6.2 *Dry gas volume.* Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using Equation 5-1.

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right) = \left(17.71 \frac{^\circ R}{in.\ Hg}\right) V_m \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}\right)$$

equation 5-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, inches Hg.

ΔH = Average pressure drop across the orifice meter, inches H_2O.

13.6 = Specific gravity of mercury.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.3 Volume of water vapor.

$$V_{w_{std}} = V_{lc}\left(\frac{\rho_{H_2O}}{M_{H_2O}}\right)\left(\frac{RT_{std}}{P_{std}}\right) = \left(0.0474 \frac{cu.\ ft.}{ml.}\right) V_{lc}$$

equation 5-2

where:

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions), cu. ft.

V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

ρ_{H_2O} = Density of water, 1 g./ml.

M_{H_2O} = Molecular weight of water, 18 lb./lb-mole.

R = Ideal gas constant, 21.85 inches Hg—cu. ft./lb-mole-°R.

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.4 Moisture content.

$$B_{ws} = \frac{V_{w_{std}}}{V_{m_{std}} + V_{w_{std}}}$$

equation 5-3

where:

B_{ws} = Proportion by volume of water vapor in the gas stream.

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions), cu. ft.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

6.5 Total particulate weight. Determine the total particulate catch from the sum of the weights on the analysis data sheet (Figure 5-3).

6.6 Concentration.

6.6.1 Concentration in gr./s.c.f.

$$c'_s = \left(0.0154 \frac{gr}{mg}\right)\left(\frac{M_n}{V_{m_{std}}}\right)$$

equation 5-4

where:

c'_s = Concentration of particulate matter in stack gas, gr./s.c.f. dry basis.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

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PLANT _____

DATE _____

RUN NO. _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER. (1 g ml):

$$\frac{\text{INCREASE, g}}{(1\ \text{g/ml})} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

6.6.3 Concentration in lb./cu. ft.

$$c_s = \frac{\left(\frac{1}{453,600}\ \frac{\text{lb.}}{\text{mg.}}\right)M_n}{V_{m_{std}}} = 2.205 \times 10^{-6}\frac{M_n}{V_{m_{std}}} \qquad \text{equation 5-5}$$

Where:

c_s = Concentration of particulate matter in stack gas (lb./cu. ft., dry basis).

453,600 = Mg/lb.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

6.7 Isokinetic variation.

$$I = \frac{T\left[\frac{V_{l_c}(\rho_{H_2O})R}{M_{H_2O}} + \frac{V_m}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{\theta V_s P_s A_n} \times 100$$

$$= \frac{\left(1.667\ \frac{\text{min.}}{\text{sec.}}\right)\left[\left(0.00267\ \frac{\text{in. Hg-cu. ft.}}{\text{ml.-°R}}\right)V_{l_c} + \frac{V_m}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{\theta V_s P_s A_n} \qquad \text{Equation 5-6}$$

where:

I = Percent of isokinetic sampling.

V_{l_c} = Total volume of liquid collected in impingers and silica gel (see Fig. 5-3), ml.

ρ_{H_2O} = Density of water, 1 g/ml.

R = Ideal gas constant, 21.83 inches Hg-cu. ft./lb.-mole-°R.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_m = Absolute average dry gas meter temperature (see Figure 5-2), °R.

P_{bar} = Barometric pressure at sampling site, inches Hg.

ΔH = Average pressure drop across the orifice (see Fig. 5-2), inches H_2O.

T_s = Absolute average stack gas temperature (see Fig. 5-2), °R.

θ = Total sampling time, min.

V_s = Stack gas velocity calculated by Method 2, Equation 2-2, ft./sec.

P_s = Absolute stack gas pressure, inches Hg.

A_n = Cross-sectional area of nozzle, sq. ft.

6.8 Acceptable results. The following range sets the limit on acceptable isokinetic sampling results:

If $90\% \leq I \leq 110\%$, the results are acceptable, otherwise, reject the results and repeat the test.

7. *Reference.*

Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.

Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0576.

Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.

Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67-119, 1967.

Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

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APPENDIX B

STACK GAS AND SAMPLING DATA

TABLE B-1. STACK GAS DATA - SECONDARY LEAD SMELTER

Run No.	$\sqrt{\Delta P}$ (avg) $\sqrt{\text{cm } H_2O}$	T_s (avg) C	P_s mm Hg	O_2, %	CO_2 %	B_{wo}, %	Md, lb/lb-mole	V_s (avg), m/s
1A	1.18	61	748.0	18.5	2.3	2.45	29.1	13.3
B						2.74		
2A	1.26	64	745.5	18.5	2.3	1.98	29.1	14.4
B						2.04		
3A	1.21	62	744.2	18.5	2.3	2.84	29.1	13.7
B						3.09		
4A	1.24	68	739.4	18.5	2.3	2.99	29.1	14.3
B						2.80		
5A	1.22	69	732.8	18.5	2.3	3.69	29.1	14.2
B						3.57		
6A	1.26	65	740.4	18.5	2.3	2.69	29.1	14.3
B						2.83		
7A	1.27	74	740.4	18.5	2.3	1.91	29.1	14.8
B						2.14		

TABLE B-2. SAMPLING DATA - SECONDARY LEAD PLANT

Run No	Meter Volume (V_m), M^3	Barometer mm Hg	ΔH mm H_2O	Avg. Meter Temp (T_m), C	Dry gas Sampled Std. Cond. (V_{mstd}), M^3	Percent Isokinetic	Average System Temperatures, C			
							Filter Box	Gas @ Filter outlet	Gas @ probe outlet	Probe Mid-point
1A	5.65	748.0	58.42	61	5.30	100	142	151	148	128
B	5.49			43	5.05	96	149	153	148	149
2A	3.79	745.5	66.04	42	3.49	99	94	98	95	94
B	3.66			46	3.33	94	97	95	93	96
3A	3.72	744.2	62.74	44	3.39	101	92	99	94	104
B	3.57			44	3.26	97	149	155	148	164
4A	3.86	739.4	66.55	45	3.50	103	94	101	94	95
B	3.69			42	3.37	99	149	157	148	138
5A	3.78	732.8	65.02	37	3.47	105	148	154	148	148
B	3.65			38	3.35	101	96	91	99	84
6A	3.64	740.4	67.31	38	3.37	102	92	94	92	108
B	3.48			36	3.25	98	151	148	148	153
7A	3.07	740.4	70.36	28	2.94	105	123	122	122	128
B	2.95			28	2.83	101	118	230	120	139